Federal Democratic
Republic of Ethiopia

☞ EDICT OF GOVERNMENT ☜

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ET 669 (2001) (English): MALTING BARLEY AND BARLEY MALT-DETERMINATION OF

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ETHIOPIAN STNADRAD

ES 669:2001

(Reaffirmed:2012)

First edition

Malting barley and barley malt ─ Determination of protein and soluble nitrogen content of wort

ICS: 67.060

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Foreword

This Ethiopian Standard has been prepared under the direction of the Technical Committee for Cereals, pulses and derived products (TC 11) and published by the Ethiopian Standards Agency (ESA).

The standard is a reaffirmation for reprint of the Ethiopian Standard ES :2001, - of , with some editorial changes without altering the technical contents in the former tex

Malting barley and barley malt—Determination of protein and soluble nitrogen content of wort

1 Scope

This Ethiopian Standard specifies the method for the determination of protein and soluble nitrogen content of wort.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Ethiopian Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Ethiopian Standard are encouraged to investigate the possibility of applying the most recent editions of the Ethiopian Standards indicated below. Registers of currently valid standards are maintained in the Quality and Standards Authority of Ethiopia.

ES ISO 6644:2001, *Cereals and milled cereal products – Automatic sampling by mechanical means.*

ES ISO 13690:2001, *Cereals, pulses and milled products – Sampling of static batches.*

3 Reagents

During the analysis, use only reagents of recognized analytical grade reagent and distilled water of equivalent purity.

a) Concentrated sulphuric acid, 98% nitrogen free;

b) Potassium sulphate or anhydrous sodium sulphate, powered;

c) Sodium hydroxide solution – 450 g of sodium hydroxide pellets or flakes shall be dissolved in 1 liter of water. It shall be boiled to remove the ammonia in a suitable stainless steel container with cover. It shall be cooled and allowed to deposit. The clear solution having specific gravity of 1.36 or higher shall then be decanted into stoppered containers;

d) Zinc granules or coarse carbonundum powder;

e) Sulphide or thisulphate solution – 40 g of commercial potassium sulphide or 80 g of sodium thiosulphate ($Na_2 S_2 O_3 . 5H_2O$) shall be dissolved in 1 litre of water.

f) Boric acid solution, 2 percent m/v in water;

g) Standard hydrochloric or sulphuric acid, 0.1N

h) Screened bromocresol green indicator – 0.1% bromocresol green in 95% ethanol shall be mixed with 0.1% methyl red in 95% ethanol in the proportions 10:4. The indicator which is pink in acid solution shall be grey at the point and blue in alkaline solution.

i) Acetanilide, pure dried in vacum at 80 °C

j) Sucrose, pure

4 Apparatus

Ordinary laboratory apparatus, and in particular the following shall be used:

a) Kjeldahl flasks 500 to 800 ml capacity

b) Kjeldahl distillation apparatus with lagged Rhodin cylindrical type spray traps, having internal tubes top and bottom, joined to a simple, tube type condenser of block tin or borosilicate glass, with the upper end stopping back to the spray and the lower end provided with a safety bulb, the tube from which dips beneath the acid in the receiving flask, as shown in the attached figure

c) Erlemeyer receiving flask – 250 ml capacity

d) Pippettes

e) Burettes

5 Sampling

Samples shall be taken in accordance with ES ISO 13690 or ES ISO 6644

6 Procedure

6.1 Duplicate samples of about 1.5 g of finely ground barley shall be taken, weighed accurately and transferred quantitatively into completely dried kjeldahl flask. About 10 g of the catalyst mixture (if tablets are used, great care must be taken to ensure that all the barley is wetted with sulphuric acid before heating) or alternatively 10 g of potassium sulphate or 0.7 g of mercuric oxide shall be added and mixed intimately with the ground barley before adding 20 ml of concentrated sulphuric acid, mixing again and digesting gently until frothing ceases. The digest shall be boiled briskly until the brown colour disappears and heating shall be continued for another 10 minutes. The digest shall be cooled and diluted gradually mixing with 250 ml of water. When mercury has been used, 25 ml of sulphide or thiosulphate solution shall be added and in all cases approximately 70 ml of strong sodium hydroxide solution shall be run underneath to provide a moderate excess over the acid to prevent bumping small pieces of zinc or coarse carbonundum are added. The flask is connected to the distillation apparatus, the sodium hydroxide solution is mixed in and the ammonia distilled into the excess of boric acid solution (about 25 ml) containing 0.5 ml of screened indicator. When the volume of the distillate has reached 180 ml, the ammonia is titrated with standard acid until the grass green colour changes to still grey, and then giving purpose colour. Paired blank determination are made to measure the correction for the reagents. It is also important to carry out a check at regular intervals according to clause 6.2 herein.

6.2 0.020 g of acetanilide and 1 g of pure sucrose are weighed and transferred to two kjeldahl flasks, A and B. The contents of each flask shall be digested and distilled into boric acid and titrated as described above. From each result, corrected by the usual blank, the nitrogen percentage of the acetanilide is calculated. This result as a percentage of 10.36 gives the percentage recovery of nitrogen, which shall be around 99.5% for flask A and at least 98% for flask B. If unsatisfactory results are obtained, tests shall immediately be made to check acid standardization and variations in digestion times and temperatures.

6.3 Determination of soluble nitrogen content soluble nitrogen content shall be determined as in clause 6.1 and 6.2 with the exceptions of the sample preparation and expression of result.

6.3.1 Preparation of sample

6.3.1.1 Take a 20 ml sample of (EBC) standard wort in a kjeldahl flask, add 2 to 3 ml of sulphric acid and evaporate this cautiously nearly to dryness, with precautions against foaming as in clause 6.1 and 6.2.

7 Expression of result

7.1 The percentage of nitrogen, on dry basis, shall be calculated as follows:-

$$N = \frac{T \times 14}{M \times D.M}$$

Where,

N is the percentage of nitrogen in dry barley,

T is the acid titration value, with the blank subtracted, corrected to extract tenth normality,

M is the mass, in grams, of barley sample taken for test and

D.M..is the percentage of dry matter

7.2 The percentage of protein, and dry basis, shall be calculated as follows:

% of protein = N x 6.25%

7.3 Soluble nitrogen content in wort shall be calculated as follows

$$N_v = \frac{T.14}{V} . 1000$$

Where

Nv is the soluble nitrogen content in wort / (mg/1)

T is acid titration value, with blank subtracted corrected to exactly 1/10 normality

14 is atomic weight of nitrogen

V is sample of volume (ml)

1000 correction factor to obtain the result per (ml/1)

Soluble nitrogen content as a percentage of dry malt obtained from:

$$N_s - \frac{N_v . E}{100{,}000 \ EW}$$

Where,

Ns is soluble nitrogen content as percentage on dry malt (%)

Nv is soluble nitrogen content (mg/1)

E' is extract of dry malt (%)

EW is grams of extract in 100 ml wort (g/100 ml)

100,000 is correction factor to obtain the result as a percentage (w/w)

7.4 Soluble nitrogen content as a percentage of the total nitrogen (kolbach index) is determined on the wort obtained from hot water extract of ES 659.

25 ml of this wort shall be taken in 500 ml kjeldhal and determined as in clause 6.1 and 6.2 herein and calculated as follows:

$$NK = \frac{N_s.100}{N}$$

Where

NK is nitrogen content as a percentage (%) (kolbach index) ;

Ns is soluble nitrogen content as a percentage of dry malt (%) ; and

N is total nitrogen content as a percentage of dry malt

8 Test result

The test report shall include the following information

a) reference to this standard ;

b) the results and method of expression used, and all information necessary for complete identification of the sample ; and

c) any unusual features noted during determination

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Ethiopian Standards Agency

Organization and Objectives

The Ethiopian Standards Agency (ESA) is the national standards body of Ethiopia established in 2010 based on regulation No. 193/2010.ESA is established due to the restructuring of Quality and Standards Authority of Ethiopia (QSAE) which was established in 1970.

ESA's objectives are:-

- Develop Ethiopian standards and establish a system that enable to check weather goods and services are in compliance with the required standards,
- Facilitate the country's technology transfer through the use of standards,
- Develop national standards for local products and services so as to make them competitive in the international market.

Ethiopian Standards

The Ethiopian Standards are developed by national technical committees which are composed of different stakeholders consisting of educational Institutions, research institutes, government organizations, certification, inspection, and testing organizations, regulatory bodies, consumer association etc. The requirements and/ or recommendations contained in Ethiopian Standards are consensus based that reflects the interest of the TC representatives and also of comments received from the public and other sources. Ethiopian Standards are approved by the National Standardization Council and are kept under continuous review after publication and updated regularly to take account of latest scientific and technological changes.
Orders for all Ethiopian Standards, International Standard and ASTM standards, including electronic versions, should be addressed to the Documentation and Publication Team at the Head office and Branch (Liaisons) offices. A catalogue of Ethiopian Standards is also available freely and can be accessed in from our website.

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